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NOTE ON ORTHO-TOLIDIN TEST FOR FREE CHLORINE

By W. F. MONFORT

Developments in the use of chlorine and hypochlorite treatment of public water supplies have enlarged the importance of methods of test for residual chlorine in the finished water, to which the paper of Ellms and Hauser (Jour. Ind. & Eng. Chem., October, 1913) is the most valuable contribution. Some experimentation by the late Dr. L. P. Kinnicutt in connection with the now celebrated or almost forgotten Boonton (N. J.) case is of considerable importance in this connection. His results are buried in the mass of reports on this case (In Chancery of New Jersey:—Jersey City vs. Jersey City Water Supply Company, Vol. 12, page 6921 et ff.) which are available to but few members of this association.

In correspondence with a number of water works chemists within the past few months it has developed that a large proportion of those addressed were unable to refer to Dr. Kinnicutt's work, and were repeating or were about to repeat unnecessarily a volume of work well done by him. It seems advisable, therefore, to make available the substance of this work, and the author asks permission to introduce it into the proceedings of this association, quoting without further comment the full concise text of his conclusions, and tabulating the experimental data on the sensitiveness of this reagent recalculated to parts per million.

The reagent as used in these experiments was prepared according to directions proposed by Prof. E. B. Phelps, as follows:

0.5 grains placed in a mortar, ground up with 10 ccm. 0.1 to 1 sulphuric acid. Transferred to a beaker, washed out into a mortar with 250 ccm. water, added 10 ccm. concentrated sulphuric acid, heated to boiling. Color deep pink, perfectly clear. Transferred to 500 ccm. tincture bottle. On standing cloudiness appeared. Filtered through asbestos.

STUDY OF THE ACTION OF CHLORINE AND OXIDIZING AGENTS ON ORTHO-TOLIDIN.
FOR THE JERSEY CITY WATER SUPPLY COMPANY. SEPTEMBER, 1909.

(OFFERED ON PAGE 6546.)

Ortho-tolidin. A homologue of benzidin, different from benzidin by the replacement of two hydrogen atoms by two methyl groups (CH_3).

(Graphic Formulae given.)

Ortho-tolidin is prepared by reduction of ortho-nitrotoluene by zinc dust in alkaline solution, and treating the azo compound formed with stannous chloride in acid solution. It is purified by recrystallization.

The reaction of various substances with benzidin have been carefully studied, but comparatively little attention has been paid to ortho-tolidin, but from our knowledge of organic compounds its behavior to various reagents, especially oxidizing agents, would be similar to that of benzidin.

In 1874, Schultz (Ann. 174, 226) showed that benzidin gave very delicate color reactions with oxidizing agents, and found that with chlorine final product of such oxidizing was a red substance insoluble in water but soluble in alcohol.

In 1881, Claus and Richter (Ber. 14, 82) studied the action of bleaching powder on ortho-tolidin and obtained an oxidation product, which however was not pure, always containing lime and also chlorine.

In 1902, Arnold and Mentzel (Ber. 35, 1328) called attention to the fact that benzidin could be used for detecting ozone, nitrogen dioxide and the halogens.

In 1904 (Ber. 37, 3761), Willstatter showed that on mild oxidation a green colored and also a yellow colored substance were formed. That the yellow compound was of a quinone nature.

In 1905 (Ber. 38, 1238), Willstatter showed that the yellow compound was formed by oxidation; also with silver oxide and lead peroxide, and that it was an oxidation product containing two less hydrogen atoms than benzidin.

The action of oxidizing agents, including chlorine, on ortho-tolidin is undoubtedly similar to their action on benzidin, and consequently, the supposition would be that the yellow substance obtained was not (as supposed) due to substitutions of chlorine, but was due to the oxidation of ortho-tolidin. In other words, that the action of bleaching powder in this case was due to potential oxygen, as is the case when bleaching powder is used in water as a germicide.

That the production of this yellow substance obtained by the action of chlorine on ortho-tolidin is due to oxidation, namely to potential oxygen, is, I think, shown by the experiments that I have made with ortho-tolidin, which are given on pages 1-17.¹ These experiments show that ortho-tolidin is acted upon not only by chlorine with the formation of a yellow substance, but by various oxidizing agents such as ozone, nascent oxygen, sodium nitrite or nitrous acid, manganese persulphate, ferric salts, potassium permanganate, potassium bichromate, lead peroxide and sodium peroxide.

That with ozone, nascent oxygen and lead peroxide the delicacy of the reaction is so great that ortho-tolidin might serve possibly better than any other reagent for detecting these substances.

That it is also a delicate reagent for potassium permanganate and potassium bichromate.

That the reaction is somewhat less marked with sodium nitrite; still less marked with ferric salts, and least marked with ammonium persulphate and sodium peroxide.

¹ Reference is to pages of Dr. Kinnicutt's notes.

That the only oxidizing agents that were tried that did not give the yellow compound with ortho-tolidin were free oxygen and hydrogen peroxide.

The reason why hydrogen peroxide did not act in the same way as other oxidizing agents may be explained by the fact that in certain cases hydrogen dioxide does not act as an oxidizing agent, as is shown, for instance, by its action on silver oxide and potassium permanganate.

That the results obtained with commercial ortho-tolidin and with chemically pure ortho-tolidin were in general the same, but that the yellow color developed quicker when using C. P. ortho-tolidin and was a purer yellow and more easily detected than when using the commercial ortho-tolidin, due to impurities in this substance.

That for testing for available chlorine in water the potassium iodide test in my experiments indicated a smaller amount of available chlorine than the commercial ortho-tolidin test. (See experiments given on page 2.)

That the C. P. ortho-tolidin did indicate as small an amount of available chlorine in water as the potassium iodide test, but the ortho-tolidin test, in my opinion, even using C. P. ortho-tolidin is not so satisfactory as the potassium iodide test, as the pale yellow color is not as satisfactory a color to the eye as a light blue color.

That the ortho-tolidin test can in no sense be considered as a specific test for chlorine.

That there is no basis, either theoretical or experimental, for the idea that the yellow compound produced by the action of potassium permanganate on ortho-tolidin is due to the entrance of manganese into the compound, and I know of no case where the hydrogen of an organic compound has been replaced by manganese.

Respectfully submitted,
LEONARD P. KINNICUTT.

SUMMARY OF TESTS OF THE SENSITIVENESS OF O-TOLIDIN WITH OXIDIZING AGENTS

| <i>Oxidizing agent</i> | <i>o-tolidin starch o-tolidin commercial iodide c.p.</i> | | | |
|-----------------------------|--|-------------------------------|-------|-------------------|
| Hypo solution..... | 0.068 | 0.017 | 0.017 | parts per million |
| Free chlorine..... | | | | |
| Ozone..... | 0.068 | 0.017 | 0.017 | very delicate |
| Nascent oxygen..... | 0.068 | 0.017 | 0.017 | very delicate |
| Sodium nitrite..... | 2.000 | | | parts per million |
| Ammonium persulphate..... | 200.000 | | | |
| Ferric chloride..... | 20.000 | | | |
| Ferric alum..... | 20.000 | | | |
| Potassium permanganate..... | 0.200 | | | |
| Potassium bichrom..... | 2.000 | | | |
| Lead peroxide..... | 3.000 | less than 3 parts per million | | |
| Sodium peroxide..... | 5000.000 | | | |